

INFLUENCE OF THE METHOD OF SYNTHESIS ON LEAD AZIDE PHOTOLYSIS

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Lead azide irrespective of method of synthesis shows the general kinetic regularities. On kinetic curves of photolysis speed $PbN_6(A6)$ characteristic sites have been defined: initial, stationary, increased and saturation. Time of the sites realization as well as the photolysis speed depends on the way of $PbN_6(A6)$ synthesis. Preliminary light processing of $PbN_6(A6)$ at $\lambda=380$ nm and $I=2 \cdot 10^{15}$ quantum \cdot sm $^{-2}$ \cdot s $^{-1}$ in vacuum ($P=1 \cdot 10^{-5}$ Pa) along with increase in photolysis speed and photocurrent in own area of absorption results in appearance of new long-wave area of spectral sensitivity. Quantum outputs and constants of photolysis speed of $PbN_6(A6)$ are determined. It is experimentally stated that the values of photocurrent observed in the field of long-wave threshold of photosensitivity coincide with the designed values of photoemission current on the border of $PbN_6(A6)$ -Pb. This fact as well as the measurements results of volt-ampere characteristics, contact photoelectrical moving force, contact potential difference reveals the formation of microheterogeneous $PbN_6(A6)$ -Pb systems (a photolysis product) at photolysis of lead. A limiting stage of $PbN_6(A6)$ photolysis is anion vacancies diffusion to neutral center of Pbn^0 .

Solid phase products released at decomposition influence significantly photochemical and photoelectrical properties of inorganic azides. Preliminary photochemical and thermal azides decomposition along with photocurrent increasing results in appearing additional maximum of photosensitivity in the area 1,65 eV on spectral-distribution curves V_ϕ and i_ϕ [1, 2]. Presence of highly-dispersed silver azide obtained as a result of influence of radioactive isotope Ag^{110} emission on salt in crystal lattice accelerates photolysis AgN_3 [3, 4]. Authors [5] notice that only minor particles of metals (decomposition products) influence catalytically. Authors [6] connect the influence of photodecomposition products on azides photolysis with electron photoemission from small metal nuclei into azide under light influence of proper wavelength with further formation of active particle (N_3°). In [7] photolysis acceleration was explained by ionization of single metal atoms. Authors [1] explain the appearance of photoelectric sensitivity of AgN_3 in spectrum long-wave region by increasing concentration of electrons photoemitted from metal in silver azide.

Systematic investigations of autocatalytic and sensitizing influence of solid phase products on inorganic azides photolysis [8–11] as well as parallel research of photolysis and electrophysical properties of artificial systems «azide – metal» [8, 12, 13] allowed advancing considerably in understanding the mechanism of inorganic azides photolysis at deep level of conversion. It is stated that physical-chemical properties (particularly thermionic work function [14]) and as a result kinetic and spectral photolysis mechanisms depend considerably on the method of lead azide synthesis [15]. In the

given report the results of work intended for investigation of synthesis method influence on kinetic and spectral photolysis mechanisms before, after and in the process of preliminary decomposition of lead azide samples, identification of solid phase product of $PbN_6(6)$ photolysis, ascertainment of energy structure of lead azide contact – photolysis product and reasons causing changes by decomposition product of photochemical and photoelectric sensitivity of lead azide are presented.

Objects and methods of investigation

Lead azide of A6 ($PbN_6(A6)$) type (in contrast to $PbN_6(AM)$ [11]) was synthesized by the method of double stream crystallization draining simultaneously 0,2 n water solutions of twice recrystallized industrial sodium azide and lead nitrate (of chemically pure type) at pH 3 and $T=293$ K during 1... 2 sec. Samples for investigations were prepared by pressing tablets $PbN_6(A6)$ with mass 150 mg at pressure $1 \cdot 10^{-3}$ kg \cdot cm $^{-2}$ or by coating 150 mg of $PbN_6(A6)$ charges on quartz plate in the form of alcohol suspension with further alcohol distillation in vacuum. Kinetic curves of photolysis rate (V_ϕ), photocurrent (i_ϕ) and photoelectromotive force (U_ϕ) of PbN_6 samples were measured at pressure $\sim 1 \cdot 10^{-5}$ Pa. Lamp RMO-4S of omegatron mass-spectrometer IPDO-1 tuned to the frequency of nitrogen registration was used as a sensor at measuring V_ϕ [11]. i_ϕ and U_ϕ were measured at the device including electrometric voltmeter V7-30 or electrometer TR-1501 [15]. Diffusion reflection (DR) spectra before and after samples irradiation were measured at pressure $\sim 10^{-4}$ Pa using the device [16] at spectrophotometer SPh-

4A with the attachment PDO-1 [17] and at pressure 101,3 kPa at spectrophotometer Specord-M40 with the attachment for reflection 8d. Mercury (DRT-250) and xenon (DKSSh-1000) lamps were used as light sources. The monochromators MDR-2 and SPM-2 and a set of optical filters were used for releasing the required spectral range of irradiation. Radiative thermoelement RT-0589 was used for actinometry of light sources. Contact potential difference (CPD) between $\text{PbN}_6(\text{A6})$ and relative platinum electrode was measured by Kelvin modified method [14]. Topography of solid phase products of lead azide photolysis was studied by coal replica technique at electron microscope UEMV-1000.

Results and discussion

Having compared kinetic regularities of lead azide photolysis and photocurrent of different methods of synthesis depending on intensity ($I=10^{13}\dots 10^{16}$ quantum $\cdot\text{cm}^{-2}\cdot\text{s}^{-1}$) and spectral composition (250...1000 nm) of incident light it was stated that lead azide reveals general kinetic regularities aside from method of its preparation. Kinetic curves V_ϕ measured at lightning $\text{PbN}_6(\text{A6})$ $\lambda=380$ nm samples at 293 K are presented in Fig. 1. It follows from the Figure that several sites may be singled out on kinetic curves V_ϕ $\text{PbN}_6(\text{A6})$ (as for $\text{PbN}_6(\text{AM})$ [11]): initial non-stationary (I), stationary (II), increased V_ϕ (III) and saturation (IV). Time of realization of kinetic curves V_ϕ different sites as well as V_ϕ values depend on the method of synthesis PbN_6 . Values of quantum yield of $\text{PbN}_6(\text{A6})$ photolysis depending on incident light intensity are presented in Table 1.

Table 1. Quantum yield of $\text{PbN}_6(\text{A6})$ photolysis

Intensity, quantum $\cdot\text{cm}^{-2}\cdot\text{s}^{-1}$	Quantum yield, 10^{-3}
$7,95\cdot 10^{14}$	1,23
$1,27\cdot 10^{15}$	1,56
$2,00\cdot 10^{15}$	1,96
$3,17\cdot 10^{15}$	3,10
$5,56\cdot 10^{15}$	5,47

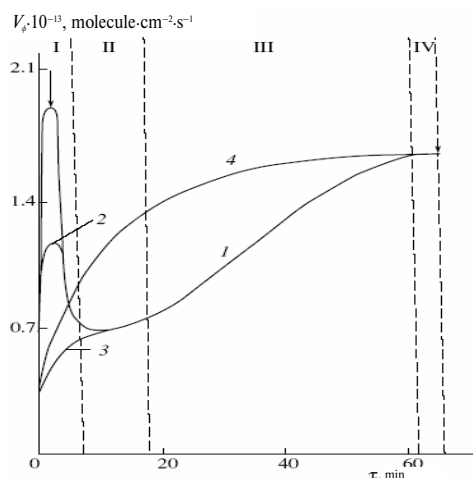


Fig. 1. Kinetic curves of $\text{PbN}_6(\text{A6})$ photolysis (V_ϕ) rate at $\lambda=380$ nm and incident light intensity $2\cdot 10^{15}$ quantum $\cdot\text{cm}^{-2}\cdot\text{s}^{-1}$ before (1) and after light interruption on I (2), II (3), IV (4) sites of kinetic curves V_ϕ . Moments of blackout are noted by arrows

Spectral-distribution curves V_ϕ and i_ϕ $\text{PbN}_6(\text{A6})$ plotted by stationary values V_ϕ and i_ϕ (site II of kinetic curves V_ϕ and i_ϕ) are presented in Fig. 2. It is seen that long-wave edge of V_ϕ and i_ϕ $\text{PbN}_6(\text{A6})$ as well as for $\text{PbN}_6(\text{AM})$ [11] is searched out at $\lambda < 410$ nm. To discover the reasons causing the observed changes of V_ϕ and i_ϕ in lightning process the experiments on influence of preliminary light treatment on kinetic and spectral dependences V_ϕ and i_ϕ were carried out. The repeated (after light interruption at I and II sites) samples lightning does not result in significant change of V_ϕ values at II, III and IV sites of kinetic curves and spectral-distribution curves V_ϕ and i_ϕ . In this case the values of V_ϕ at site I decrease (Fig. 1 curves 2 and 3). After preliminary lightning of $\text{PbN}_6(\text{A6})$ samples during 60 min ($\text{PbN}_6(\text{AM})$ lightning during 5 min [11]) the form of kinetic curves (Fig. 1 curve 4) V_ϕ and spectral-distribution curves $\text{PbN}_6(\text{A6})$ V_ϕ and i_ϕ (Fig. 2) changes significantly. Along with increasing V_ϕ and i_ϕ in proper absorbing region of $\text{PbN}_6(\text{A6})$ new photosensitivity regions which long-wave thresholds extend to 600 and 850 nm for $\text{PbN}_6(\text{AM})$ [11] and $\text{PbN}_6(\text{A6})$ correspondingly appear on spectral-distribution curves V_ϕ and i_ϕ . Longer (during 3 h) light influence of $\lambda=380$ nm and intensity $2\cdot 10^{15}$ quantum $\cdot\text{cm}^{-2}\cdot\text{s}^{-1}$ on $\text{PbN}_6(\text{A6})$ samples and on $\text{PbN}_6(\text{AM})$ samples (during 40 min [11]) results in decreasing V_ϕ and i_ϕ . As a result of electron microscope and spectrophotometric investigations it was stated that the observed decrease of lead azide photosensitivity is connected with sample surface blackout with solid phase photolysis product and as a result decreasing the number of light quanta absorbed by lead azide.

$V_\phi \cdot 10^{-13}$, molecule $\cdot\text{cm}^{-2}\cdot\text{s}^{-1}$

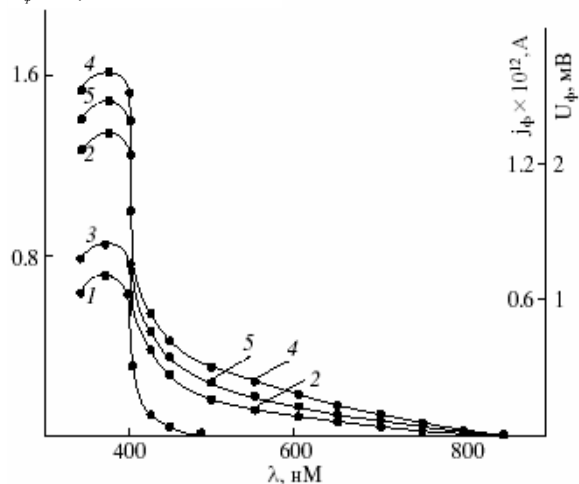


Fig. 2. Spectral distribution of V_ϕ (1,5), i_ϕ (2,3) and U_ϕ (4) before (1, 2) and after (3-5) $\text{PbN}_6(\text{A6})$ light irradiation $\lambda=380$ nm and $I=2\cdot 10^{15}$ quantum $\cdot\text{cm}^{-2}\cdot\text{s}^{-1}$

When finishing lightning on different sites of kinetic curves V_ϕ the site of postgasrelease is observed. It is stated that independently of preliminary exposure time the curves of postgasrelease are rectified in coordinates $\ln C_{\text{N}_2} = f(t)$. Values of rate constants (k) after light interruption on different sites of kinetic curves V_ϕ were estimated by slope ratio of dependence $\ln C_{\text{N}_2} = f(t)$ (Table 1).

Table 2. Rate constants (k , s^{-1}) of the process responsible for postgasrelease (site IV) after light interruption at I, II and IV sites of kinetic curve V_ϕ

Sample	Rate constant, s^{-1}		
	Site I	Site II	Site IV
PbN ₆ (A6)	$(2,2 \pm 0,11) \cdot 10^{-2}$	$(3,7 \pm 0,12) \cdot 10^{-2}$	$(3,0 \pm 0,15) \cdot 10^{-3}$

The results of investigations of heavy metal azide photolysis obtained in the given paper and earlier [1–14] indicate the fact that the main reason of the observed (due to light treatments) changes of kinetic and spectral curves V_ϕ and i_ϕ is the formation of lead azide photolysis on lighted surface of samples of solid phase product.

To identify the solid phase photolysis product PbN₆(A6) the approach proposed in [18–20] was used. Authors of [18–20] suggested comparing experimentally the observed photocurrent dependence on emission frequency with photoemission current on the boundary «metal-dielectric» for finding metal particles in dielectrics. The authors of [18–20] obtained full agreement of theory and experiment by the example of silver bromide and chloride.

Photoemission current on the boundary PbN₆(A6) – Pb caused by monochromatic light of frequency $\omega > \omega_0$ where ω_0 is the red boundary of photoeffect, was calculated by [18–20]:

$$I = A(\omega - \omega_0)^2 f(\gamma),$$

$$f(\gamma) = \int_0^1 \frac{2(1-x)dx}{1 - \exp[-(\gamma x)^{-1/2}]} =$$

$$= \left\{ \begin{array}{l} 1 + 8\gamma \exp(-\gamma^{-1/2}) + \dots, \gamma \ll 1, \\ \frac{8}{15}\gamma^{1/2} + \frac{1}{2} + \frac{2}{9}\gamma^{-1/2} + \dots, \gamma \gg 1 \end{array} \right\},$$

where A is the constant determined by metal and boundary properties; x is the integration variable $\gamma = h(\omega - \omega_0)/E_A$ is the characteristic parameter; h is the Planck constant; $E_A = 33,5\varepsilon^2 m/m_0$ is the characteristic energy; m_0 is the electron mass, m is the effective mass; ε is the dielectric permeability of medium.

Calculated values of photoemission current on the boundary of PbN₆(A6) – Pb and i_ϕ dependence on quantum energy of incident light measured for PbN₆(A6) samples subjected to preliminary lightning $\lambda = 380$ nm and $I = 2 \cdot 10^{15}$ quantum·cm⁻²·s⁻¹ during 60 min are compared in Fig. 3. It follows from Fig. 4 that calculated values of photoemission current on the boundary PbN₆(A6) – Pb and experimentally observed photocurrent values practically coincide. As a result of measuring CPD between relative platinum electrode and PbN₆(A6) and lead artificially coated on the surface of lead azide tablets it was stated that photochemical decomposition at light influence ($\lambda = 380$ nm, $I = 2 \cdot 10^{15}$ quantum·cm⁻²·s⁻¹) to the site IV of kinetic curves V_ϕ and i_ϕ in fine vacuum environment results in increasing CPD values for PbN₆(A6) (Table 3) and CPD values for the samples subjected to photolysis coincide satisfactorily with values measured for artificially coated lead ([14], Table 3).

When investigating topography of solid phase product of lead azide photolysis it is stated that particles mainly of size 30...50 and 90...110 Å of spherical form

at incident light intensity $I = 4 \cdot 10^{14} \dots 8 \cdot 10^{15}$ quantum·cm⁻²·s⁻¹ and times of samples irradiation corresponding to reaching the sites I and II of kinetic curve V_ϕ are formed. Quantity of particles and their size increase as incident light intensity and exposure time correspondingly growth.

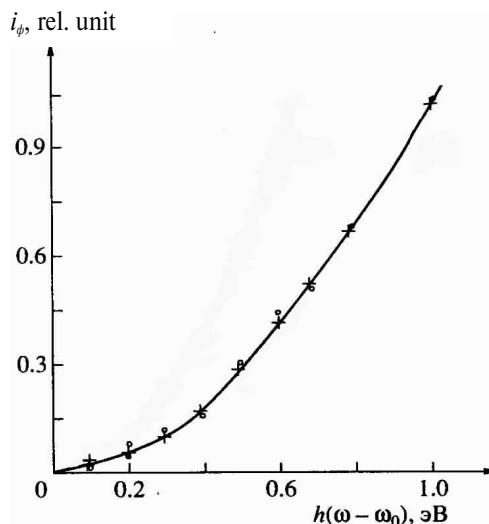

Fig. 3. Comparison of calculated (x) values of photoemission current and experimental values of photocurrent (o) in relative units on the boundary of PbN₆(A6)-Pb

Table 3. Contact potential difference (B) between PbN₆(A6), Pb and relative platinum electrode at 293 K

Sample	Pressure, Pa			
	$1 \cdot 10^5$	$1 \cdot 10^{-5}$	$1 \cdot 10^{-5*}$	$1 \cdot 10^{-5**}$
PbN ₆ (A6)	-0,34	-0,21	+0,58	+0,59
Pb	+0,58	+0,59	+0,59	

*After preliminary thermal treatment at 550 K during 180 min
 **After preliminary photolysis at $\lambda = 380$ nm, $I = 2 \cdot 10^{15}$ quantum·cm⁻²·s⁻¹ during 90 min

Long-wave edge of PbN₆(A6) DR (as well as PbN₆(Am) [11]) is at $\lambda = 410$ nm. Samples treatment with light $\lambda = 380$ nm in the range of intensities $I = 4 \cdot 10^{14} \dots 8 \cdot 10^{15}$ quantum·cm⁻²·s⁻¹, along with absence of noticeable effects in proper absorption domain of PbN₆(A6) results in significant change of spectral curves form of DR in area $\lambda \geq 410$ nm. At irradiation times corresponding to I and II sites realization on kinetic curves V_ϕ along with DR decrease in the range of 400...800 nm on spectral curves of DR maximums at $\lambda \approx 440$ nm and $\lambda \approx 600$ nm appears. Further increase of light treatment time to the site (III) results in bands broadening and maximums shift to the long-wave spectrum area. The results of comparison of areas (S) dependences corresponding to the change of samples reflectance calculated by DR spectra at different times and intensities of incident light with the number of photolytic metal atoms (N) calculated by kinetic curves V_ϕ are presented in Fig. 4. Values of rate constants of PbN₆(A6) photolysis are given in Table 4.

It is seen from the presented table that constants of PbN₆(A6) photolysis rate calculated by kinetic curves of photolysis rate (k_{ph}) and DR spectra (k_{DR}) coincide satisfactorily. Experimental facts presented in the given paper and earlier indicate the fact that metal lead is the solid phase product of PbN₆(A6) photolysis (as well as PbN₆(Am) [11]).

Table 4 Constants of $PbN_6(A\bar{6})$ photolysis rate calculated by kinetic curves of photolysis rate (k_{ph}) and spectra of diffuse reflection (k_{DR})

Intensity, quantum·cm ⁻² ·s ⁻¹	$k_{ph} \cdot 10^2$	$k_{DR} \cdot 10^2$
$7,95 \cdot 10^{14}$	4,15-0,24	4,22-0,32
$1,27 \cdot 10^{15}$	4,75-0,48	4,52-0,61
$2,00 \cdot 10^{15}$	4,87-0,27	4,67-0,44
$3,17 \cdot 10^{15}$	5,93-0,56	5,69-0,61
$5,56 \cdot 10^{15}$	3,50-0,23	3,66-0,17

To discover the mechanism of lead (photolysis product) influence on the process of $PbN_6(A\bar{6})$ photolysis and as a result on changing kinetic curves and spectral-distribution curves V_ϕ and i_ϕ (due to preliminary samples treatment $\lambda=380$ nm, $I=2 \cdot 10^{15}$ quantum·cm⁻²·s⁻¹) volt-ampere characteristics (VAC) and U_ϕ characteristics of $PbN_6(A\bar{6})$ – Pb systems (photolysis product) were measured.

It was stated from VAC analysis and CPD measuring results (Table 2, [14]) that in the contact region of $PbN_6(A\bar{6})$ – Pb (due to unconformity between operation of electron output from contacting partners) double electric layer appears. Contact of $PbN_6(A\bar{6})$ – Pb reveals rectifying properties (external stress impressed in the direction opposite to CPD corresponds to straight direction, that is plus of the source is supplied to $PbN_6(A\bar{6})$). Contact of $PbN_6(A\bar{6})$ – Pb does not reveal rectifying properties [11]. It is seen from Fig. 2 that U_ϕ polarity being constant along the whole spectrum corresponds to the positive sign from lead azide side and spectral-distribution curves U_ϕ , V_ϕ , i_ϕ correlates with each other.

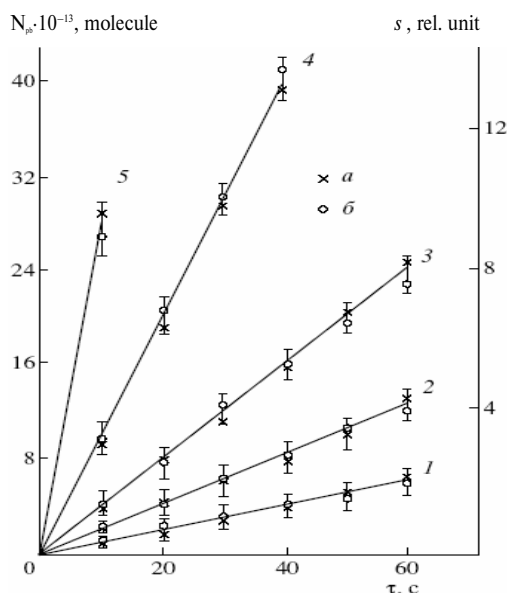


Fig. 4. Comparison of photolytic lead amount N (\times) and areas S (\circ) corresponding to the changes of $PbN_6(A\bar{6})$ diffuse reflection depending on incident light intensity: 1) $7,95 \cdot 10^{15}$; 2) $1,27 \cdot 10^{15}$; 3) $2,00 \cdot 10^{15}$; 4) $3,17 \cdot 10^{15}$; 5) $5,56 \cdot 10^{15}$ quantum·cm⁻²·s⁻¹

The data obtained in the given paper and earlier [8, 9, 11, 13, 14] indicate first of all the fact that the main products of $PbN_6(A\bar{6})$ photolysis at 293 K in fine vacuum environment ($P=1 \cdot 10^{-5}$ Pa) are metal lead and gaseous nitrogen. U_ϕ generation as well as rectifying effects at VAC indicate directly the formation of micro-heterogeneous systems $PbN_6(A\bar{6})$ – Pb in the process of

lead azide photolysis. On their boundaries dark and photo processes evidently ensure change of V_ϕ and i_ϕ in the proper absorption domain of lead azide as well as appearance of new long-wave regions of photosensitivity (Fig. 1, 2). Photochemical occurrence of photoelectric processes in such systems may be caused by redistribution of charge carriers generated by light under the influence of contact field [21]. These processes result in considerable changes of photolysis behavior in preliminary photo decomposed preparations of lead azide. The diagram of energy bands of heterosystems $PbN_6(A\bar{6})$ – Pb is presented in Fig. 5. When constructing the diagram measuring results of CPD, VAC, U_ϕ , V_ϕ , i_ϕ spectral distribution data as well as measuring results of external photo effect from the observed objects were used [22].

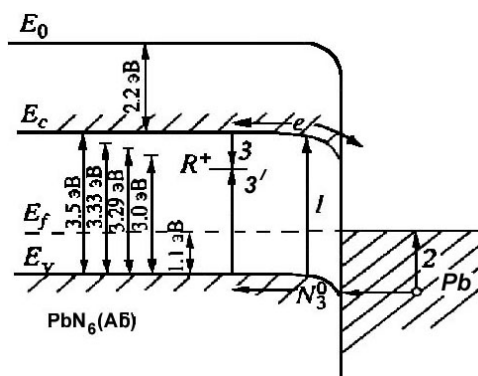
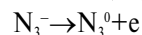


Fig. 5. Diagram of energy bands of $PbN_6(A\bar{6})$ -Pb systems. E_v is the level of valence band top, E_c is the level of bottom of conduction band, E_f is the Fermi level, E_0 is the vacuum level, R^+ is the recombination centre

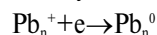
The intensive generation of electron hole drops in lead azide occurs at light influence from the proper absorption domain of lead azide (Fig. 5, transition 1)



As quantum yield of photolysis estimated by the initial region of kinetic curve V_ϕ is amounted to 0,002...0,01 then a part of photoinduced charge carriers is recombined (Fig. 5, transitions 3)



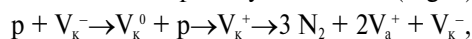
where R^+ is the recombination centre they are also redistributed in contact field with nonequilibrium electrons transition from conductivity zone of lead azide to lead



In this case U_ϕ is formed with positive sign from the side of lead azide (Fig. 3) which may promote further increasing particles sizes

$Pb_n^0 + V_a \rightarrow [Pb_n^0 V_a] + e \rightarrow [Pb_n^0 V_a e] + V_a \rightarrow [Pb_n^0 2V_a e] + e \rightarrow Pb_{n+1}^0$, where V_a is the anionic vacancy (lead azide is disordered by Schottky [23]).

As the particle size of photolytic lead increases the hole amount in the area of space charge of lead azide growths. The resulting growth of hole concentration results in both i_ϕ and V_ϕ increasing by the accepted reactions for lead azide photolysis – site III (Fig. 1).



where V_a^+ and V_k^- are the anionic and cation vacancy correspondingly.

Photoemission of holes from lead to valence zone of lead azide occurs at the influence of light from long-wave region of spectrum on heterosystems $\text{PbN}_6(\text{A6}) - \text{Pb}$ (Fig. 5, transition 2). It results in appearance of U_ϕ , V_ϕ and i_ϕ in preliminary photo decomposed preparations in spectrum long-wave region. The revealed regularities of changing lead azide photosensitivity with photolytic lead in a long-wave region coincide with the above-stated. Really, U_ϕ of positive sign from the side of lead azide is formed (Fig. 2), energy position of long-wave threshold of U_ϕ , V_ϕ and i_ϕ for the systems $\text{PbN}_6(\text{A6}) - \text{Pb}$ coincides satisfactorily with the value of energy barrier for holes transition from metal into valence band of lead azide (Fig. 5, transition 2). Activation energy of photolysis of systems $\text{PbN}_6(\text{A6}) - \text{Pb}$ in the long-wave region of spectrum ($E_a=0,51$ eV) coincides satisfactorily with the value of photolysis activation energy in proper absorption region of $\text{PbN}_6(\text{A6})$ ($E_a=0,48$ eV) and for systems $\text{PbN}_6(\text{AM}) - \text{Pb}$ differs per a value of energy threshold for electron transition from valence band $\text{PbN}_6(\text{AM})$ into metal ($E_a=0,65$ eV) and amount $E_a=1,2$ eV [11].

To determine a limiting stage of the growth process of photolytic lead particles the time during which the mobile anionic vacancy neutralizes electron or diffuses to the neutral centre was estimated. The relaxation time

by the mechanism of anionic vacancies drift in Coulomb field to the localized electron equals to the maxwellian relaxation time [24]

$$\tau_i = \varepsilon / 4n\sigma,$$

where ε is the dielectric permeability ($\varepsilon_{\text{PbN}_6}=6$), σ is the specific conductivity at 293 K ($\sigma_{\text{PbN}_6} \approx 1 \cdot 10^{-12} \text{ Ohm}^{-1} \cdot \text{cm}^{-1}$), $\tau_i=0,4$ s. Constant of photolysis rate was amounted to $k^1=2,5 \text{ s}^{-1}$.

The average relaxation time at diffuse process occurring may be estimated [24]

$$\tau_0 = e^2 / \sigma k_b a T,$$

where e is the electron charge; a is the lattice constant ($a_{\text{PbN}_6}=8 \cdot 10^{-10} \text{ cm}$); $T=293$ K, k_b is the Boltzmann constant. At $T=293$ K $\tau_0=80$ s. The constant of photolysis rate (k^1) amounts in this case to $k^1 \approx 1,25 \cdot 10^{-2} \text{ s}^{-1}$.

Satisfactorily coincidence of photolysis rate constants (Table 4) and constants of process rate responsible for post-gas-release (Table 2) with k^1 leads to the conclusion that the limiting stage of $\text{PbN}_6(\text{A6})$ photolysis process is the diffusion of anionic vacancies to the neutral centre.

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